Pub. No.:	WO/1999/029388	International Application No.:	PCT/AT1998/000299
Publication Date:	17.06.1999	International Filing Date:	09.12.1998

IPC: *B01D 15/08* (2006.01), *B01J 8/02* (2006.01), *B01J 8/04* (2006.01), *G01N 30/58* (2006.01)

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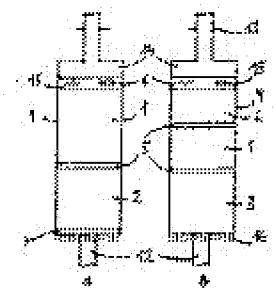
Priority Data: A 2076/97 09.12.1997 AT

Title: (EN) ANNULAR CHROMATOGRAPH
(DE) ANNULARCHROMATOGRAPH

Abstract:

(EN) The invention relates to an annular chromatograph with a particle bed in its annular gap. Said chromatograph is characterised in that at least one reaction zone for conducting the chemical reactions and at least one associated separation zone for the chromatographic separation are provided.
(DE) Die Erfindung liefert einen Annularchromatographen mit einem Teilchenbett in seinem Ringspalt, der dadurch gekennzeichnet ist, daß zumindest eine Reaktionszone zur Durchführung von chemischen Reaktionen mit zumindest einer zugehörigen Trennzone zur chromatographischen Trennung vorgesehen ist.



Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW.
African Regional Intellectual Property Org. (ARIPO) (GH, GM, KE, LS, MW, SD, SZ, UG, ZW)
Eurasian Patent Organization (EAPO) (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM)
European Patent Office (EPO) (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE)
African Intellectual Property Organization (OAPI) (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Publication Language: German (DE)

Filing Language: German (DE)

Annularchromatograph the instant invention concerns a Annularchromatographen with a particle bed in its annular gap.

Annularchromatographie represents since some years recognized and in always strong increased measures practiced variant of preparative chromatographic separations. Preferably Annularchromatographie becomes used, if large amounts at substance mixtures are to be isolated, since this type of the chromatography can become continuous operated, and with relative high scale.

A typical P-CAC-apparatus ("P-CAC", preparative continuous annular chromatography = preparative continuous Annularchromatographie) consists of an annular, i.e. "annularem", particle bed, which is into the space (annular gap) between two concentric cylinders packed. Bottom rotation of the

particle bed around its axis at the upper end continuous feed solution as well as or several Eluenten is given up. Such procedures are widespread to the state of the art known and (e.g. see. EP-A-371.648).

In the preparative chemistry, in particular on the field of the biochemistry and the medical chemistry, the degree of purity of the products of chemical reactions of critical importance, why frequent most expensive prepurifying, processing and final purification procedures to the use come, is to be lowered in order the content at inevitable impurities on if possible low values, ideally the bottom detection limit.

Such cleaning methods become majority in separated apparatuses and in the batch operating conducted, i.e. the purification of the starting products and the final product made e.g. chromatographic in Vorsäulen and/or. in downstream separation sow len, which a continuous operation prevented, there the separating agents after made separation of the substance mixtures only again purged, regenerated and/or. equilibrated to become to have. In the best case thus an intermittent feed stream is in semikontinuierlicher procedure guidance possible. Thus however the flow rate of such plants limited, the machine, temporal and financial effort for such methods as well as in the sequence is also the price for kind of such of prepared products high.

An other problem, with which practitioners are confronted with chromatographic separations, is the compromise between the time interval required for the separation, i.e. the residence time substances in the column, and the resolution of the substance mixtures achieved thereby. General one sinks the residence time - however (mostly) also the resolution - with rising flow rate of the Eluens and reverse, whereby the achievement good separative power generally relative low flow rates become prefered.

In the contrast in addition normally high throughputs and thus also high flow rates become desired for chemical reactions in flow reactors, which opposes the continuous operation of reaction and separation additional.

Target of the invention lay therefore in the provision of a annularchromatographischen plant for the continuous execution of chemical reactions and pre and/or. downstream cleaning steps in continuous and thus wirtschaftl icherer procedure. An other target was the optimization of the residence times of the substances in the single portions of the plant.

This target becomes according to invention by a Annularchromatographen with a particle bed in its annular gap achieved, which is characterized by the fact that at least a reaction zone is provided for the execution of chemical reactions with at least an associated separation zone to the chromatographic separation. Such an arrangement of reaction and separation zone in a single annularchromato graphic column (in arbitrary order) possible successive reaction (EN) and separation (EN) and/or. Prepurifying (EN) and reaction (EN) in the perfect continuous operation, i.e. not mere semikontinuierlich bottom intermittent feed of the feed stream, there with Annularchromatographen that or the desired products at desired local positions along the column extent from the column and/or. occur in the

present case from the corresponding zone out and into the next zone. An other advantage of this system lies in the fact that withdrawn in such Reaktionschromatographen the reaction products formed in the reaction zones become continuous from the reaction zone, which shifts the reaction equilibrium to the product side, from which rapid and generally quantitative conversions result.

To the separation of reaction mixtures and separation obtained in such a "fixed bed reactor" and/or purification of reaction products formed in a reaction zone is at least according to invention a reaction zone above at least a separation zone disposed. Alternative ones or additional in addition reaction zone can be (n) the chemical (n) reaction (EN), running off, prefered likewise according to invention at least a separation zone above at least a reaction zone disposed for prepurifying at least a starting product for in that at least.

Also combinations of reaction and separation zones in almost arbitrary number and order are possible according to invention. So for example several reaction zones can do a rear and/or after a separation zone for prepurifying or. it follows among themselves that again or several separation zones to the resolution of the reaction products and - by-products attach. In this way are feasible even multistage reactions as well as extremely specific and selective and separations in a single reactor/a chromatograph.

The material for the separation zone (n) can become thereby from anion exchange resins, cation exchange resins, exclusion gels, gel permeation gels, affinity gels, hydrophobic one chromatography (H IC) gels, displacement (DISPLACEMENT) resins, reverse phases (Reversed phase) gels and electrophoresis gels or arbitrary other usually separating agents selected used in chromatographic separation methods. Arbitrary combinations of such separation gels can come and - resins to the use depending upon separation problem. With use of electrophoresis gels will in each case at the upper and bottom edge of the electrophoretic separating layer electrodes disposed, in order to apply voltage. The electrical terminal made thereby for example over Schleifring-Kontakt to rotational axis the column. Details for this are in the likewise appended, at the 1. December 1997 filed österr.

Patent application of the applicant with the announcing number A 2030/97.

The material for the reaction zone (n) can do general from the same materials as the separation zone (n) as well as out opposite the reactions inert material, depending upon type of reaction, running off therein, e.g. Glass beads, activated carbon, (if necessary modified) polymers, alumina, silica gel, etc., selected become. Prefered according to invention is glass beads as well as activated carbon. The material for the reaction zone (n) knows however in preferable embodiments of the invention with or several reaction catalysts, like e.g. Metallic one, metal complexes or enzymes, impregnated and/or. coated its, e.g. Pd/C, Pt/C etc. This measure possible for example the common supply of several reaction partners in a single feed stream, whereby it comes only with contact with a catalyst immobilized in the reaction zone to the actual reaction.

Alternative one in addition knows the material for those at least a reaction zone prefered likewise according to invention with at least reactants coated its, i.e. or several other reactants can together with catalyst (EN) in a feed stream supplied become, the reaction comes it however again only in the reaction zone in the chromatograph; in addition, according to invention can become all reactants at the particle material of the reaction zone immobilized and only that or the required (n) catalyst (EN) with the feed fed, if or several Components of the feed stream at least reactants of the solid phase displace to bring around this with that or the remaining reactants in contact.

The particle bed in the Annularchromatographen according to invention can consist of a single material or different materials for reaction and separation zone, whereby the reaction zone material if necessary like described above impregnated and/or. coated to be can ignore and the two particle materials if necessary continuous into one another. In a preferable embodiment however all zones contained in the chromatograph are spatial from each other separated by separating layers, in order to prevent a mixture both the particle materials and the single streams between the respective zones. Such separating layers know thereby from membranes, non-porous, inert particle material and -- particular with application of electrophoresis - from electric non conductive material selected its. Prefered ones become also here glass beads, which are non conductive both for majority of the reactions inert and electric coming into question.

In an other preferable embodiment the particle bed is with a cover layer covered and/or is underlaid with a base layer, whereby Deckund consists the base layer preferably of the same material as the separating layer (EN), in particular glass beads. Is for example the highest and/or. lowest zone to the electrophoretic separation certain, is planning a covering and/or. To recommend base layer in each case, in order to hold the electric field if possible constant.

In the Annularchromatographen is usually the particle bed in the barrel of uniform thickness, which has to the sequence that the flow rate of the liquid phase essentially constant and/or. by the packing density of the particle bed certain is. According to invention will proposed to train the annular gap in the Annularchromatographen over its height with zones various thickness whereby adapting zones can be present between them which cause as stepless a flow change as possible and usually curved or conical are. In preferable embodiments/is however at least in a part of the height of the particle bed of the inner cylinders and/or the outer cylinder of the reactor, preferably conical, is on that in each case different tapered formed, which the flow rate in the particle bed increased and by shortening of the Laufstrecke of the solutes and corresponding limitation of the diffusion and migration the same in the particle bed in the sequence a reduction of the gang-wide and thus a Konzentrationsetfekt effected.

/In other embodiments of the inner cylinders and/or the outer cylinder of the reactor can be able to do alternative ones or additional in addition at least over a part of the height of the particle bed, preferably conical, of in each

case different the formed to be running away. Thus the flow rate in the particle bed becomes reduced, which e.g. in separation zones an increase of the resolution to the sequence has.

The constrictions are corresponding, i.e. the sites with gathering the Säulenwänden, prefered according to invention at the end of reaction zones with subsequent separation zone, in order to give the feed up at the start of the separation zone as concentrated ones as possible, while extensions are to be preferably found within the range of separation zones, where them -- like mentioned above -- the separative power of the column continue to improve.

The formation of the constrictions and/or. Extensions as cone the ensured uniformity of the current in these ranges, whereby it can hardly come also at these sites to undesirable jam, Nebenströmen or back mixtures.

In preferable embodiments a keeping at a moderate temperature coat provided is, over the solutions transported in the column heats up and/or at the chromatograph according to invention in the range at least a zone at the interior and/or outer cylinder. to be able cool. This can in particular be in reaction zones of importance, where a certain reaction temperature is to be kept, in addition, it can the chromatographic separation in separation zones by the temperature affected become, why also keeping at a moderate temperature coats are appropriate on both for reaction and separation zones in the scope of the invention.

In other embodiments outer cylinder is a radiation source as heat source and/or as reaction catalyst and/or in the range at least a zone at Innenund/or. - initiator provided. That is, that for example not only heating of certain ranges of the chromatograph by means of IR or microwave radiation, but also the release of photochemical reactions (by means of e.g. visible or UV light) within the column made to become to be able.

A more detailed description of the invention bottom reference follows after the accompanying designs, where Fig. la) and lb) schematic views of embodiments of the Annularchromatographen of the invention are; Fig. 2a) and 2b) schematic views of other embodiments of the Annularchromatographen of the invention are; Fig. 3 a schematic sectional view of an embodiment of a Annularchromatographen according to invention with radiation source and keeping at a moderate temperature coat is; Fig. 4 a schematic sectional view of a Annularchromatographen according to invention with passage area modifications is; and Fig. ä) - 5f) sketches of possible embodiments of the Annularchromatographen according to invention with constrictions and/or. Extensions of the liquid flow shows.

Fig. 1 shows schematic two embodiments of the instant invention, i.e. a Annularchromatographen with a reaction zone 1 and one and/or. two separation zones 2, 3 in a annularen column out opposite the components Reaktionsund separation solutions inert material, preferably glass, existing from an inner cylinder 8 and an outer cylinder 9 (whereby in Fig. 1 only the outer cylinder 9 to see is). (Of a not represented engine propelled) the column is 2 rotatable stored around an axis 1 and becomes over leads 1 3 for feed and solvent, an header 14 as well as supply channels 15 continuous fed.

The channels 15 know the conventional arrangement forms, i.e. Single, repeated or Slot nozzles or such a thing, exhibit, for the invention prefered will however to the column extent adapted curved slot nozzles different width, in order to be able to co-ordinate the feed and Eluentenströme as precise ones as possible one on the other.

At the lower end the columns are discharge channels and/or. - tube 16 to the collection of the eluates provided. These outlets 16 can be either with the column connected (i.e. they rotate with these around axis 12) or however at the axis 12 fixed its and e.g. over a slip ring with the relative column in contact, rotary in addition, stands itself, whereby latter embodiment becomes prefered. The particle material of the highest zone 1 and/or. 2 is in each case with a cover layer 6 covered, into which the supply channels 15 preferably dive, in order to ensure uniform object. In Fig. Ia) an additional base layer is 7 shown, (additional not porous bottom plate represented to one -, e.g. Frit, diaphragm disk, etc.) serves to prevent a withdrawing of particle material at the bottom of the column. The single reaction and separation zones are usually 5 separated by separating layers, in order to prevent a mixing of the particle materials of the two zones.

The material for the separation, the covering and the base layers 5, 8, 9 will selected from membranes as well as non-porous, opposite all components of the respective reaction and separation solutions inert particle material and can for all three layers same or different be, whereby it may not be however particular conductive electric for electrophoretic separations. Prefered according to invention is glass beads, since these with practical all common applications inert and light are to be applied.

In Fig. Ia) a single reaction zone 1 and a separation zone are 2 provided. The material for the reaction zone can from arbitrary particle materials inert opposite the reactions running off therein, like e.g. Glass beads, preferably such with diameters of approximately 150-240 pm, as well as from material with separation efficiency, like e.g. Ionenaustauschharzen, exclusion resins, etc., selected its, whereby that Particle material in the reaction to participate can (e.g. more Ionentauscher, h catalysis o.a.) or not. The material of the reaction zone 1 can also with or several reactants and/or catalyst (e.g. Metal complexes, enzymes, pH modifiers etc.) coated its, so that the reaction at the solid phase runs off. Can even be immobolisiert theoretical all reactants at the carrier, if at least a component supplied together with the feed solvent (e.g. the solvent) at least a reaction partner from the connection to the solid phase displaced, i.e. from this strips.

With operation of such Annularchromatographen a feed solution, which contains at least one reactants and/or catalyst, over the supply channels 1 5 into the column fed and arrived from there into the reaction zone 1, where it comes to the desired chemical reaction of the reaction partners. These occur 5 and subsequent the separation zone 2 at the lower end the zone 1 the separating layer, where it comes to the separation and purification of the substance mixture.

The so separated components, product (e), catalyst, output and possible byproduct (e) step at the lower end the column over the discharge opening tubes 16 at defined site (i.e. at a certain angular position) along the periphery of the Annularchromatographen from the system and are caught there and if necessary in tanks and/or. Processing plants (to restricting, precipitation, etc.) passed on.

The height and the diameter of the single zones become by the type of the reaction and the separation, which intended residence time of the substances in the column, which type of the particle materials, which packing density of the respective zones, which desired resolution of the separation and other factors affected, which are specialists on the field known throughout. The average formed skilled person is in the layer, the dimensioning the corresponding specific problem definition, e.g. empirical or by preliminary tests to make.

In Fig. Ib) three 5 zones 1, 2, 3 from each other separated by separating layers are provided. Of it the zones 2 and 3 are designed as separation zones and the zone located between them 1 as reaction zone. Thus or several components of the feed solution in the separation zone 2, fed over the supply tubes 1 5, can be prepurified, before in zone 1 the desired reaction can run off. Subsequent one follows in similar manner as bottom reference after Fig. Ia) a described separation of the reaction products in separation zone 3.

In Fig. 2a) and 2b) are other embodiments of the invention schematically shown. In Fig. 2a) in each case two separation and reaction zones are 2, 3 and/or. 1, 4 shown. In such Annularchromatographen a multistage synthesis can become continuous conducted, whereby following a first reaction step in reaction zone 1 an intermediate cleaning in separation zone 2, then a second reaction step in reaction zone 4 and the final purification in separation zone 3 conducted to finally become to be able.

Fig. 2b) an embodiment with a reaction and two shows separation zones 1 and/or.

2, 3, where a mixture in two stages, outgoing from reaction zone 1, can become purified, which highly pure products at the column discharge opening 16 possible.

In Fig. 3 is particularly a partial section of one preferable embodiment of the invention schematically shown. Two separation and reaction zones each provided, a zone 2 for prepurifying, are i.e. similar Fig. Ib), two successive reaction zones 1, 4 for the execution of a two-stage synthesis, as well as again a separation zone 3 for a final cleaning step.

The first reaction zone 1 is here provided with a radiation source 11, which is 9 disposed along the inner periphery of the inner cylinder 8 and the outside periphery of the outer cylinder, in order to be able to expose the total volume of the zone to if possible same the moderate radiation. As radiation any type of electromagnetic radiation comes into question, e.g. visible light and UV light as reaction catalysts, IR and microwave radiation as heat source; prefered become UVund microwave radiation.

Subsequent one follows an other reaction zone 4 for the second reaction step, which with a keeping at a moderate temperature coat, i.e. However heating or cool one coat, provided is, which serves either to bring the reaction mixture on the required reaction temperature or (as in this case), in order to cool it down after the radiation effect in zone 1 before the subsequent separation. Such keeping at a moderate temperature coats can become naturally also at separation zones mounted, in order to keep at a moderate temperature the separation mixtures direct, i.e. mostly to cool.

The power supply of the radiation source and the keeping at a moderate temperature coat can from the inside -- over the axis 12 -- or take place from the outside.

Although into the Fig. 1 to 3 maximum in each case two reaction and separation zones shown are, are contained any other number and sequence of such zones convertible into the practice in the scope of the invention.

To the regulation of the flow rate of the mobile phase in the single zones provisions can be met to the modification of the flow area. A constriction of the same effected for example a more rapid current in this portion and thus - as already described - a concentration effect, while an extension of the flow area has slowed current and thus better interaction with the stationary phase to the sequence. In a reaction zone an improvement of the separative power results, i.e. from such an extension to more complete conversion, in a separation zone. Resolution.

Fig. a possible modification at the transition of a reaction zone 1 points 4 to a separation zone 2. At the lower end the zone 1 constricted itself the cross section of the column, in the fig on 1/4 of the original value, which to an increase of the flow rate (here e.g. on the 1 6fache) and thus to a concentration of the mixture outgoing from the reaction zone, that leads a separating layer 5 in the sequence, e.g. from glass beads, that flows through among other things a range with constricted cross section however parallel cylinder walls covers, which becomes 7 referred as concentration zone 1. Finally the mixture occurs the separation zone 2. During the transition of the separating layer 5 into the separation zone 2 extended itself the cross section (and the flow rate sinks) again on the initial value, which becomes interactions with the solid phase again stronger and the resolution of the mixture into its components thus improved. An extension of the cross section on higher values than in zone 1 would bring an other improvement to the separative power with itself. The formation of the constrictions and/or. Extensions as cone the ensured uniformity of the current in these ranges, whereby it can hardly come also at these sites to undesirable jam, Nebenströmen or back mixtures, since flow turbulences become minimized in this way.

General one is however a compromise between the residence time of the mixture in the column, i.e. the flow rate of the column on the one hand and the conversion and/or. the resolution on the other hand to be received, in order to optimize a Annularchromatographen according to invention for a

given reaction/separation system, i.e. the flow area cannot become arbitrary large or small made.

The ratio between the maximum and the minimum annular gap-wide preferably lies between 10: 1 and 1,5: 1, in particular between 5: 1 and 1,5: 1. The height of the concentration zones 1 7 and/or. The minimum-wide of the annular gap respective value can preferably extend zones with improved resolution up to 2/3 of the bed depth over one, whereby the maximum-wide of the annular gap respective value becomes particularly prefered.

Fig. shows 5 schematic various embodiments of the column cross section, whereby the Fig. 5a) and 5e) a constriction and/or. Extension, those represent by inclination only to a cylinder wall (alternatively interior or outer cylinders) to the other one and/or. from this away formed become. The Fig. 5b) and 5f) show a constriction in analogous manner and/or. Extension by reciprocal inclination of the cylinder walls, and in the Fig. 5c) and 5d) are an in and/or. a reciprocal constriction with preceding concentration zone 1 7 shown. From this - like also already from Fig. 4 - that also several constrictions and/or extensions sequences can, over the liquid flow stepwise is to be recognized on very rapid and/or. slow flow rates high and/or. to down-regulate, if this is helpful to the respective requirements at the Annularchromatographen of the invention.

The number of the application possibilities of the Annularchromatographen according to invention is almost unlimited, why here only in the approach and general on various homo and heterogeneous catalysis processes, most diverse hydrogenations, dehydrogenations, redox reactions, hydraulic and other Solvolysen, enzyme reactions u.v.m. one refers. A concrete example is the hydrolysis and subsequent separation of oligomerer carbohydrates, e.g. by acidic catalysis:
EMI13.1

< SEP> H'
< tb> Raffinose < SEP> t < SEP> D-fructose < SEP> + < SEP> D glucose < SEP> + < SEP> D-galactose
< tb> or by enzymatic cleavage:
EMI13.2

< SEP> (I-CD (D < .tOSIdJSC
< tb> Raffinose < SEP> t < SEP> D-fructose < SEP> + < SEP> D glucose < SEP> + < SEP> D-galactose
< tb>

CLAIMS: 1. Annularchromatograph with a particle bed in its annular gap, characterised in that at least a reaction zone (1, 4) for the execution of chemical reactions with at least an associated separation zone (2, 3) to the chromatographic separation provided is.

2. Annularchromatograph according to claim 1, characterised in that at least a reaction zone (1, 4) above at least a separation zone (2, 3) to the separation and/or purification of reaction products disposed formed in that at least reaction zone (1, 4) is.

3. At least Annularchromatograph according to claim 1 or 2, characterised in that for prepurifying at least a starting product for (n) the chemical (n) reaction (EN), running off in that at least reaction zone (1, 4), a separation zone (2, 3) above at least a reaction zone (1, 4) disposed is.

4. Annularchromatograph after one of the claims 1 to 3, characterised in that the material for the separation zone (n) (2, 3) from anion exchange resins, cation exchange resins, exclusion gels, gel permeation gels, affine one itätsgelen, hydrophobic chromatography (H IC) gels, displacement (DISPLACEMENT) resins, reverse phases (Reversed phase) gels and electrophoresis gels selected is.

5. Annularchromatograph after one of of the preceding claims, characterised in that the material for the reaction zones (1, 4) from the same materials as the separation zone (n) (2, 3) as well as out opposite the reactions running off therein inert material, e.g. Glass beads or activated carbon, selected is.

6. Annularchromatograph after one of the preceding claims, characterised in that the material for those at least a reaction zone with reaction catalyst, like e.g. Metallic one, metal complexes or enzymes, impregnated and/or. coated is.

7. Annularchromatograph after one of the preceding claims, characterised in that the material for those at least a reaction zone with at least reactants coated is.

8. Annularchromatograph after one of the preceding claims, characterised in that all zones (1, 2, 3, 4) by separating layers (5) spatial from each other separated are.

9. Annularchromatograph after one of the preceding claims, characterised in that those at least a separating layer (5) from membranes, non-porous, inert particle material, and electric non conductive material, preferably glass beads, selected is.

10. Annularchromatograph after one of the preceding claims, characterised in that the particle bed with a cover layer (6) covered and/or with a base layer (7) is underlaid, whereby the covering and the base layer (6, 7) preferably from the same material as the separating layer (EN) (5) exists.

11. Annularchromatograph after one of the preceding claims, characterised in that at least over a part of the height of the particle bed of the inner cylinders (8) and/or the outer cylinder (9) of the reactor to the increase of the flow rate in the particle bed, preferably conical or curved, on which in each case different tapered formed is/is.

1 2. Annularchromatograph after one of the preceding claims, characterised in that at least over a part of the height of the particle bed of the inner cylinders (8) and/or the outer cylinder (9) of the reactor to the reduction that Flow rate in the particle bed, preferably conical or curved, of in each case different the formed is running away/is.

1 3. Annularchromatograph according to claim 11 or 12, characterised in that of the inner cylinders (8) and/or the outer cylinder (9) of the reactor in the

lower end region at least a reaction zone (1, 4), preferably conical, on which in each case different tapered formed is/is.

14. Annularchromatograph after one of the claims 11 to 13, characterised in that of the inner cylinders (8) and/or the outer cylinder (9) of the reactor in the range at least a separation zone (2, 3), preferably conical, of in each case different the formed is running away/is.

15. Annularchromatograph after one of the preceding claims, characterised in that in the range at least a zone (1, 2, 3, 4) at the interior and/or outer cylinder (8, 9) a keeping at a moderate temperature coat (10) provided is.

1 6. Annularchromatograph after one of the preceding claims, characterised in that in the range at least a zone (1, 2, 3, 4) at the interior and/or outer cylinder (8, 9) a radiation source (11) as heat source and/or as reaction catalyst and/or. - is initiator provided.